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FORM-PTO	0-1390 U.S. DEI	PARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER
(1107. 5 20		R TO THE UNITED STATES	009760-015
	DESIGNATED/ELEC	CTED OFFICE (DO/EO/US)	U.S. APPLICATION NO. (If known, see 37 C F.R. 1.5)
	CONCERNING A FILI	NG UNDER 35 U.S.C. 371/	Պ ա ա ա ա ա ա ա ա ա ա ա ա ա ա ա ա ա ա ա
	ATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
	200/04671	12 July 2000	16 July 1999
	FINVENTION MOPLASTIC RESIN COMPOS	SITION AND MOLDED ARTICLE THEREOF	-
	ANT(S) FOR DO/EO/US	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	
KEIICH	II KANAKA, TOSHIO SHIWA	KU, and MINEO OHTAKE	
Applicar	nt herewith submits to the United S	tates Designated/Elected Office (DO/EO/US) the follow	ving items and other information:
1.	This is a FIRST submission of ite	ems concerning a filing under 35 U.S.C. 371.	
2.	This is a SECOND or SUBSEQUE	NT submission of items concerning a filing under 35 U	I.S.C. 371.
3.	This is an express request to beg (9) and (21) indicated below.	gin national examination procedures (35 U.S.C. 371(f))	. The submission must include Items (5), (6),
4.	The US has been elected by the	expiration of 19 months from the priority date (Article	31).
5.	A copy of the International Appl	ication as filed (35 U.S.C. 371(c)(2))	
	a. 🛛 ` is attached hereto (red	quired only if not communicated by the International Bi	ureau).
	b. 🛛 has been communicat	ed by the International Bureau.	,
	c. \Box is not required, as the	application was filed in the United States Receiving O	ffice (RO/US).
6.	An English language translation	of the International Application as filed (35 U.S.C. 371	(c)(2))
	a. 🛛 is attached hereto.		
	b. D has been previously s	ubmitted under 35 U.S.C. 154(d)(4).	
7. 🗆 🖂	Amendments to the claims of th	e International Application under PCT Article 19 (35 U	.S.C. 371(c)(3))
	a. are attached hereto (r	equired only if not communicated by the International	Bureau).
	b. D have been communication	ated by the International Bureau.	
	c. have not been made;	however, the time limit for making such amendments	has NOT expired.
	d. 🛛 have not been made a	and will not be made.	
8. 🗆	An English language translation	of the amendments to the claims under PCT Article 19	(35 U.S.C. 371(c)(3)).
9.	An oath or declaration of the inv	rentor(s) (35 U.S.C. 371(c)(4)).	
10.	An English language translation 371(c)(5)).	of the annexes to the International Preliminary Examına	ation Report under PCT Article 36 (35 U.S.C.
Items 1	1 to 20 below concern document(s) or information included:	
11. 🖾	An Information Disclosure State	ment under 37 CFR 1.97 and 1.98.	
12.	An assignment document for red	cording. A separate cover sheet in compliance with 37	CFR 3.28 and 3.31 is included.
13.	A FIRST preliminary amendment		* (
14.	A SECOND or SUBSEQUENT pre	eliminary amendment.	A.
15. 🗆	A substitute specification.		
16.	A change of power of attorney a	and/or address letter.	
17. 🗆	A computer-readable form of the	e sequence listing in accordance with PCT Rule 13ter.2	2 and 35 U.S.C. 1.821 - 1.825.
18.	A second copy of the published	international application under 35 U.S.C. 154(d)(4).	
19. 🗆	A second copy of the English la	nguage translation of the international application unde	r 35 U.S.C. 154(d)(4).
20.	Other items or information:		
	certified copy of Japanese Patent A	Application 11-202839, filed 16 July 1999, was submi ity has been perfected.	tted during the international phase of the



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21. 🛛	The following	fees are submitted:			CALC	CULATIONS	PTO USE ONLY
Basic Nati	ional Fee (37 C	FR 1.492(a)(1)-(5)):					<u> </u>
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and	all claims satist	fied provisions of PCT Article	33(1)-(4)	\$100.00 (962)			
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	is enclosed.						
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	(703) 836	a, Virginia 22313-1404 i-6620		NAME			
				22,030 REGISTRATION NUMBER		January [*] DATE	15, 2002

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Patent Attorney's Docket No. <u>009760-015</u>

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re F	Patent Application of)	
)	BOX PCT
KEIIC	HI KANAKA et al.)	
)	Attention: DO/EO/US
Applic	eation No.: (Unassigned))	
)	
Filed:	January 15, 2002)	Group Art Unit: (Unassigned)
)	
For:	THERMOPLASTIC RESIN)	Examiner: (Unassigned)
	COMPOSITION AND MOLDED)	
	ARTICLE THEREOF)	

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents Washington, D.C. 20231

Sir:

This is a national phase filing of International Application No. PCT/JP00/04671, filed July 12, 2000.

Please amend the Application as follows:

IN THE CLAIMS:

Kindly replace Claims 6 to 11 as follows:

6. (Amended) The composition according to claim 1, wherein the phosphorus oxoacid monoester and diester (C) are phosphonates represented by the following formula (III):

$$H(OH)_{m}P(=O) (OR)_{2-m}$$
 (III)

wherein m is 0 or 1 and R is a monovalent organic group.

7. (Amended) The Composition according to claim 1, wherein the phosphorus oxoacid monoester and diester (C) are phosphonates represented by the following formula (IV):

$$HP (= O) - Y \qquad (IV)$$

wherein Y is a divalent α , ω -dioxy organic group.

- 8. (Amended) The composition according to claim 1, which further contains a thermoplastic resin not forming an anisotropic molten phase, except for (A) and (B), in an amount of 1 to 90 parts by weight to 100 parts by weight of the total amount of (A) and (B).
- 9. (Amended) The composition according to claim 1, which further contains in inorganic filler in an amount of 1 to 100 parts by weight to 100 parts by weight of the total amount of (A) and (B).
- 10. (Amended) A molded article prepared by molding the composition according to claim 1:
- 11. (Amended) An injection molded article, wherein the liquid crystal polymer (B) capable of forming an anisotropic molten phase is present in the state of fibers having

an average aspect ratio of 5 or more in a matrix of the thermoplastic polyester resin (A) not forming an anisotropic molten phase as a result of an injection molding of the composition according to claim 1.

Kindly add the following new Claims 12 to 20:

12. (New) The composition according to claim 2, wherein the phosphorus oxoacid monoester and diester (C) are phosphonates represented by the following formula (III):

$$H(OH)_{m}P(=O) (OR)_{2-m}$$
 (III)

wherein m is 0 or 1 and R is a monovalent organic group.

13. (New) The composition according to claim 3, wherein the phosphorus oxoacid monoester and diester (C) are phosphonates represented by the following formula (III):

$$H(OH)_{m}P(=O) (OR)_{2-m}$$
 (III)

wherein m is 0 or 1 and R is a monovalent organic group.

14. (New) The composition according to claim 4, wherein the phosphorus oxoacid monoester and diester (C) are phosphonates represented by the following formula (III):

$$H(OH)_{m}P(=O) (OR)_{2-m}$$
 (III)

wherein m is 0 or 1 and R is a monovalent organic group.

15. (New) The Composition according to claim 2, wherein the phosphorus oxoacid monoester and diester (C) are phosphonates represented by the following formula (IV):

$$HP (= O) - Y \qquad (IV)$$

wherein Y is a divalent α , ω -dioxy organic group.

16. (New) The Composition according to claim 3, wherein the phosphorus oxoacid monoester and diester (C) are phosphonates represented by the following formula (IV):

$$HP (= O) - Y \qquad (IV)$$

wherein Y is a divalent α , ω -dioxy organic group.

17. (New) The composition according to claim 2, which further contains a thermoplastic resin not forming an anisotropic molten phase, except for (A) and (B), in an amount of 1 to 90 parts by weight to 100 parts by weight of the total amount of (A) and (B).

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Application No. (Unassigned)
Attorney's Docket No. 009760-15
Page 5

- 18. (New) The composition according to claim 2, which further contains in inorganic filler in an amount of 1 to 100 parts by weight to 100 parts by weight of the total amount of (A) and (B).
- 19. (New) A molded article prepared by molding the composition according to claim 2.
- 20. (New) An injection molded article, wherein the liquid crystal polymer (B) capable of forming an anisotropic molten phase is present in the state of fibers having an average aspect ratio of 5 or more in a matrix of the thermoplastic polyester resin (A) not forming an anisotropic molten phase as a result of an injection molding of the composition according to claim 2.

REMARKS

The present Amendment modifies the claim format only so as to eliminate the use of multiple dependence.

An Information Disclosure Statement is being filed concurrently herewith.

The examination and allowance of the Application are respectfully requested.

Respectfully submitted,

BURNS, DOANE, SWECKER & MATHIS, L.L.P.

Bv:

Benton S. Duffett, Jr. Registration No. 22,030

P.O. Box 1404 Alexandria, Virginia 22313-1404 (703) 836-6620

Date: January 15, 2002

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531 Rec'd PCT/PT: 15 JAN 2002

Application No. (Unassigned) Attorney's Docket No. 009760-15 Mark-up of Claims - Page 1 of 1

Attachment to Preliminary Amendment dated January 15, 2002 Mark-Up of Claims 6, 7, 8, 9, 10 and 11

6. (Amended) The composition according to [any of claims 1 to 4] <u>claim 1</u>, wherein the phosphorus oxoacid monoester and diester (C) are phosphonates represented by the following formula (III):

$$H(OH)_{m}P(=O) (OR)_{2-m}$$
 (III)

wherein m is 0 or 1 and R is a monovalent organic group.

7. (Amended) The Composition according to [any of claims 1 to 5] <u>claim 1</u>, wherein the phosphorus oxoacid monoester and diester (C) are phosphonates represented by the following formula (IV):

$$HP (= O) - Y \qquad (IV)$$

wherein Y is a divalent α , ω -dioxy organic group.

8. (Amended) The composition according to [any of claims 1 to 7] claim 1, which further contains a thermoplastic resin not forming an anisotropic molten phase, except for (A) and (B), in an amount of 1 to 90 parts by weight to 100 parts by weight of the total amount of (A) and (B).

Application No. (Unassigned)
Attorney's Docket No. 009760-15
Mark-up of Claims - Page 2 of 2

Attachment to Preliminary Amendment dated January 15, 2002 Mark-Up of Claims 6, 7, 8, 9, 10 and 11

- 9. (Amended) The composition according to [any of claims 1 to 8] <u>claim 1</u>, which further contains in inorganic filler in an amount of 1 to 100 parts by weight to 100 parts by weight of the total amount of (A) and (B).
- 10. (Amended) A molded article prepared by molding the composition according to [any of claims 1 to 9] claim 1:
- 11. (Amended) An injection molded article, wherein the liquid crystal polymer (B) capable of forming an anisotropic molten phase is present in the state of fibers having an average aspect ratio of 5 or more in a matrix of the thermoplastic polyester resin (A) not forming an anisotropic molten phase as a result of an injection molding of the composition according to [any of claims 1 to 9] claim 1.

531 Rec'dPC 15 JAN 2002

Description

THERMOPLASTIC RESIN COMPOSITION AND MOLDED ARTICLE THEREOF

Technical Field of the Invention

The present invention relates to a resin composition blended with a thermoplastic polyester resin not forming an anisotropic molten phase and a liquid-crystal polymer, and a molded article thereof.

Background Art

A liquid-crystal polymer (LCP) capable of forming an anisotropic molten phase is a thermoplastic resin having numerous properties such as high strength, high rigidity, high thermal resistance, mold processability. However, it makes a difference in molding shrinkage and mechanical properties between a molecular chain orientation direction and a transverse direction, and further, it has commercial disadvantages such as high cost.

On the other hand, a thermoplastic polyester resin such as polycarbonate resin (PC), which dose not form an anisotropic molten phase, is relatively inexpensive, but there is a disadvantage that it is inferior to LCP in the physical properties such as heat resistance and rigidity.

Especially, since the resin lacks enough rigidity to use for a thin-film housing, it must be thickened on a design.

Therefore, there is a limit in its applicability to recent miniaturization/lightening in the fields of electric, electronic, and communication apparatus.

Many trials have, then, been made to make the most of the advantages of LCP and thermoplastic resin not forming an anisotropic molten phase while making up the disadvantages of both by mixing and using them. However, an injectionmolded article of a resin composition obtained by simply blending a thermoplastic resin with a small amount of LCP, cannot utilize the LCP's properties such as high strength, high rigidity, thermal resistance and 'moldability, and results in remarkable deterioration of the mechanical strength. This is because the exhibition of LCP's high mechanical properties and the like originates from a molecular orientation formed under shear and stretch stresses during melt processing. In the resin composition obtained by simply blending a thermoplastic resin and a small amount of LCP, although molecules are particularly oriented in the vicinity of the surface layer of the molded article, most of LCP, other than the surface layer, are only spherically dispersed in a matrix of the thermoplastic resin and provided with no reinforcing effect. Specifically, it is not until LCP takes the form of fibers inside the molded

article that LCP molecules are oriented in the length direction of a fibrous configuration, so that the resin is provided with the reinforcing effect.

Moreover, when the proportion of LCP increases and the thermoplastic resin not forming an anisotropic molten phase is reduced, LCP this time becomes a matrix in which the thermoplastic resin is dispersed as islands. Such the resin composition is of little utility value since the advantages of the thermoplastic resin cannot be utilized.

Accordingly, methods of producing a molded article containing fibrous LCP provided with a reinforcing effect have been proposed as described in JP-A 5-70700 and JP-A 5-112709, wherein a molding material is first prepared beforehand by stretching and extruding LCP and a thermoplastic resin such as PC at a temperature at which both are molten so that LCP is present as fibers having a high aspect ratio, and then, during molding to form the molded article, the molding material is molded at a temperature at which LCP fails to be molten and only the thermoplastic resin such as PC is molten.

In these methods, however, LCP is stretched and extruded in advance to be oriented as fibers. Alternatively, to form the molded article without any preparation, LCP needs to be formed into fibers by applying a considerable shearing force when a mold is filled up with the resin

composition. The former case supposedly results in deteriorated fluidity and limited molding conditions. In the latter case, there are a remarkable influence of the molded article's shape and a possibility of insufficient strength due to partially insufficient fibrous forming.

In consideration of the aforementioned problems, a resin composition has been discovered in which, by blending a specific phosphorus compound such as a pentaerythritoltype triester phosphite, it is no longer necessary to extrude LCP in advance so that it is oriented in a fibrous state, LCP is easily modified into a fibrous state to exhibit a reinforcing effect in molding at such a temperature that the LCP is allowed to flow in a sufficient degree, and thereby the mechanical strength, heat resistance, molding-easiness, etc. of the obtained molded article record quite high values closer to those of LCP (JP-A 7-179743). This resin composition is a material having excellent properties as a molding material for a thin-wall molded article. The most distinct characteristic of this resin composition lies in that LCP is easily modified into a fibrous state by injection molding as described above, an extremely high reinforcement effect which has never been observed is obtained, and therefore the resulting molded article has unique properties and can be a thin-wall one especially excellent in mechanical strength.

However, in the resin composition, there arises a problem that the excellent properties of the molded article expected as described above, are unstable due to a variation of melt-kneading conditions in producing the molding material such as the degree of exhaust from a vent and the degree of contact with ambient air. Therefore, an additive which achieves an excellent effect in a more stable manner has been required.

In consideration of the aforementioned problems, the inventors of the present invention have assiduously studied to obtain a resin composition which can stably exhibit excellent properties as a molding material. As a result, they have discovered that, by injection molding a resin composition, in which one or more kinds of compounds selected from phosphorus oxoacid monoesters and diesters has been added to a resin component comprising a liquid crystal polymer and a thermoplastic polyester resin which does not form an anisotropic molten phase, LCP is easily modified into a fibrous state in the molded article and exhibits an extremely high reinforcing effect which has never been observed, and therefore the resulting molded article has unique properties and an excellent mechanical strength, and these characteristics are stably exhibited in the controllable production conditions of the resin composition. Thus, the present invention has been accomplished.

Disclosure of the Invention

That is, the present invention relates to a thermoplastic resin composition, prepared by blending 100 parts by weight in total consisting of 99 to 50 parts by weight of a thermoplastic polyester resin (A) which does not form an anisotropic molten phase and 1 to 50 parts by weight of a liquid crystal polymer (B) capable of forming an anisotropic molten phase, and 0.001 to 2.0 parts by weight of one or more kinds of compounds (C) selected from phosphorus oxoacid monoesters and diesters represented by the following formulae (I) and (II):

$$(X)_{n}P(=O)(OR)_{3-n}$$
 (I)

$$(X)_{n}P(OR)_{3-n} \qquad (II)$$

wherein n is 1 or 2; X is a hydrogen atom, a hydroxyl group or a monovalent organic group and, when there are plural X's, they may be the same or different; and R is a monovalent organic group and, when there are plural R's, they may be the same or different.

Namely, a composition comprising (A), (B) and (C) at a ratio as above shown.

The component (C) may include phosphonates represented by the following formula (IV):

$$HP(=O)-Y \qquad (IV)$$

wherein Y is a divalent α , ω -dioxy organic group.

Particularly, a characteristic of the present invention is to provide a molded article excellent in mechanical strength on which fibrously oriented LCP acts as reinforcing component by using the phophorus oxoacid monoesters and diesters.

Detailed Description of the Invention

The present invention will be described in detail hereinafter.

Examples of the thermoplastic polyester resin (A) not forming an anisotropic molten phase which is used in the present invention include polyesters obtained by polycondensation of a dicarboxylic acid compound and a dihydroxy compound, polycondensation of oxycarboxylic acid compounds, or polycondensation of a mixture of these three components, and either homopolyesters or copolyesters can be effective on the present invention. Examples of the dicarboxylic acid compound herein include an aromatic dicarboxylic acid, an alicyclic dicarboxylic acid, an aliphatic dicarboxylic acid, a heterocyclic dicarboxylic acid, a multifunctional carboxylic acid, and ester-forming

derivatives thereof such as alkyl esters and phenyl esters of the above-mentioned carboxylic acids. Specifically, terephthalic acid, isophthalic acid, phthalic acid, naphthalenedicarboxylic acid, biphenyldicarboxylic acid, stilbenedicarboxylic acid, 2,2-(biscarboxyphenyl)propane, biscarboxyphenyl sulfone, cyclohexanedicarboxylic acid, succinic acid, adipic acid, sebacic acid, dodecanoic diacid, dimer acid, pyridinedicarboxylic acid, trimellitic acid, trimesic acid, pyromellitic acid, and ester-forming derivatives thereof are cited. These compounds may be used singly or as a combination of two or more compounds. Examples of the dihydroxy compounds used herein include ethylene glycol, propane diol, butane diol, hexane diol, neopentyl glycol, cyclohexane diol, cyclohexanedimethanol, diethylene glycol, triethylene glycol, hydroquinone, bisphenol A, bisphenol S, biphenol, 2,2-(bishydroxyethoxyphenyl)propane, bishydroxyethoxyphenyl sulfone, 4,4-bis(hydroxyethoxy) biphenyl, dimerdiol, polyethylene glycol, polytetramethylene glycol, trimethylolpropane, pentaerythritol and ester-forming derivatives thereof. They may be used singly or as a combination of two or more compounds. Examples of the oxycarboxylic acid include hydroxybezoic acid, hydroxynaphthoic acid, 4-hydroxy-4'-carboxybiphenyl and ester-forming derivatives thereof. They may be used singly

or as a combination of two or more compounds.

Another example of the thermoplastic polyester resin

(A) not forming an anisotropic molten phase which is used in the present invention, is a polycarbonate resin having a repeating unit represented by the following formula (V):

$$-(-Q-A-Q-O-C-O-)_n-$$
 (V)

wherein Q is selected from aromatic groups such as naphthylene, phenylene, halogen-substituted phenylene and alkylene group-substituted phenylene; A is selected from $-CR_1R_2$ -, >C=0, -O-, -S-, -SO- and $-SO_2$ -, in which R_1 and R_2 are selected from H, a hydrocarbon group and a group which forms a cycloalkane group with adjacent carbon atoms.

Specifically, the polycarbonate resin is a polycarbonate based polymer produced by reacting one or more kinds of bisphenol compounds with phosgene or diester carbonate. Examples of the bisphenol compounds include: bis(hydroxyaryl)alkanes such as bis(4-hydroxyphenyl)methane, 1,1-bis(4-hydroxyphenyl)ethane, 1,1-bis(4-hydroxyphenyl)propane, 2,2-bis(4-hydroxyphenyl)propane, 2,2-bis(4-hydroxyphenyl)propane, 2,2-bis(4-hydroxyphenyl)pentane, 2,2-bis(4-hydroxyphenyl)octane, 2,2-bis(4-hydroxyphenyl)-3-methylbutane, 2,2-bis(4-hydroxyphenyl)hexane, 1,1-bis(4-hydroxyphenyl)cyclopentane, 1,1-bis(4-hydroxyphenyl)cyclopentane, 1,1-bis(4-hydroxyphenyl)cyclopentane, 1,1-bis(4-

hydroxyphenyl)cyclohexane, bis(4-hydroxy-3methylphenyl) methane, 1,1-bis(4-hydroxy-3methylphenyl)ethane, 2,2-bis(4-hydroxy-3methylphenyl)propane, 2,2-bis(4-hydroxy-3,5dimethylphenyl)propane, 2,2-bis(4-hydroxy-3ethylphenyl)propane, 2,2-bis(4-hydroxy-3-tbutylphenyl)propane, 2,2-bis(4-hydroxy-3-bromophenyl)propane, bis (4-hydroxyphenyl) phenylmethane, bis (4-hydroxyphenyl) diphenylmethane, bis (4-hydroxyphenyl) dibenzylmethane, 1,1-bis (4hydroxyphenyl)-1-phenylpropane, and 2,2,2',2',-tetrahydro 3,3,3',3'-tetramethyl-1,1'-spirobi-[1H-indene]-6,6'-diol; dihydroxyaryl ketones such as 4,4'-dihydroxydiphenyl ketone, 4,4'-dihydroxy-3,3'-dimethyldiphenyl ketone; dihydroxyaryl ethers such as 4,4'-dihydroxydiphenyl ether, 4,4'-dihydroxy-3,3'-dimethyldiphenyl ether; dihydroxyaryl sulfides such as 4,4'-dihydroxydiphenyl sulfide, 4,4'-dihydroxy-3,3'dimethyldiphenyl sulfide; dihydroxyaryl sulfones such as 4,4'-dihydroxydiphenyl sulfone, 4,4'-dihydroxy-3,3'dimethyldiphenyl sulfone; dihydroxyaryl sulfoxides such as 4,4'-dihydroxydiphenyl sulfoxide, 4,4'-dihydroxy-3,3'dimethyldiphenyl sulfoxide.

Among them, one or more kinds of resins selected from a polycarbonate resin, a polyalkyleneterephthalate resin and a polyarylate resin is especially preferably employed as the

thermoplastic polyester resin (A) not forming an anisotropic molten phase which is used in the present invention. More preferably sekected is one or more kinds of resins selected from a polycarbonate resin and a polyarylate resin, and most preferably, a polycarbonate resin.

The polyalkylene terephthalate resin may be polyethylene terephthalate and/or polybutylene terephthalate resin.

The liquid-crystal polymer (LCP) (B) capable of forming an anisotropic molten phase used in the present invention refers to a melt-processed polymer with the property that it can form an optically anisotropic molten The properties of an anisotropic molten phase can be confirmed by the conventional polarization inspection method using orthogonal polarizers. More specifically, confirmation of anisotropy in a molten phase can be attained by using a Leitz polarization microscope and observing a molten sample mounted on a Leitz hot stage under the nitrogen atmosphere at a magnification of 40 times. The LCP applicable to the present invention exhibits an optical anisotropy wherein a polarized light penetrates even in a static molten phase, when observed between cross polarizers. As the LCP usable in the present invention, aromatic polyester and aromatic polyester amide are preferable, and polyester partially containing these structures in the same

molecular chain is also preferable.

Examples of the components or monomer groups constituting such LCP include:

- (1) an aromatic hydroxycarboxylic acid, an aromatic aminocarboxylic acid and derivatives thereof;
- (2) an aromatic dicarboxylic acid, an alicyclic dicarboxylic acid and derivatives thereof;
- (3) an aromatic diol, an alicyclic diol, an aliphatic diol and derivatives thereof; and
- (4) an aromatic hydroxyamine, an aromatic diamine and derivatives thereof. By using these components, the LCP is constituted, for example, of solely (1), or a combination of, for example, (2) and (3), (2) and (4), (1), (2) and (3), (1), (2) and (4), and (1), (2), (3) and (4).

Examples of the aromatic hydroxycarboxylic acid and the aromatic aminocarboxylic acid (1) include 4-hydroxybenzoic acid, 3-hydroxybenzoic acid, 6-hydroxy-2-naphthoic acid, 6-hydroxy-1-naphthoic acid, 4-(4-hydroxyphenyl)benzoic acid, 3-methyl-4-hydroxybenzoic acid, 3,5-dimethyl-4-hydroxybenzoic acid, 2,6-dimethyl-4-hydroxybenzoic acid, 3-methoxy-4-hydroxybenzoic acid, 3,5-dimethoxy-4-hydroxybenzoic acid, 3-phenyl-4-hydroxybenzoic acid, 6-hydroxy-5-methyl-2-naphthoic acid, 6-hydroxy-5-methoxy-2-naphthoic acid, 2-chloro-4-hydroxybenzoic acid, 3-chloro-4-hydroxybenzoic acid, 3,5-dichloro-4-hydroxybenzoic

acid, 2,5-dichloro-4-hydroxybenzoic acid, 2,6-dichloro-4-hydroxybenzoic acid, 3-bromo-4-hydroxybenzoic acid, 6-hydroxy-5-chloro-2-naphthoic acid, 6-hydroxy-7-chloro-2-naphthoic acid, 4-aminobenzoic acid, 3-aminobenzoic acid, 6-amino-2-naphthoic acid and the like.

Examples of the aromatic dicarboxylic acid and the alicyclic dicarboxylic acid (2) include terephthalic acid, isophthalic acid, 2,6-naphthalenedicarboxylic acid, 2,7-naphthalenedicarboxylic acid, 1,4-naphthalenedicarboxylic acid, 1,5-naphthalenedicarboxylic acid, 4,4'-diphenylcarboxylic acid, 3,4'-diphenylcarboxylic acid, 3,3'-diphenylcarboxylic acid, 4,4"-terphenyldicarboxylic acid, diphenylether-4,4'-dicarboxylic acid, diphenoxyethane-4,4'-dicarboxylic acid, diphenoxyethane-4,4'-dicarboxylic acid, diphenoxyethalic acid, dimethylterephthalic acid, phenylterephthalic acid, methoxyterephthalic acid, chloroterephthalic acid, dichloroterephthalic acid, bromoterephthalic acid, 1,4-cyclohexanedicarboxylic acid, 1,3-cyclohexanedicarboxylic acid, [2.2.2]bycyclooctanedicarboxylic acid.

Examples of the aromatic diol, alicyclic diol and aliphatic diol (3) include hydroquinone, resorcinol, 2,6-naphthalene diol, 1,5-naphthalene diol, 2,7-naphthalene diol, 4,4'-dihydroxydiphenyl, 3,3'-dihydroxydiphenyl, 3,4'-dihydroxydiphenyl, 4,4"-dihydroxyterphenyl, 4,4'-dihydroxydiphenyl ether, 2,2-bis(4-hydroxyphenyl)propane,

4,4'-dihydroxyphenyl ketone, 4,4'-dihydroxydiphenyl sulfide,
4,4'-dihydroxydiphenyl sulfoxide, N-(4-hydroxyphenyl)-3hydroxyphthalimide, chlorohydroquinone, methylhydroquinone,
2,5-dimethylhydroquinone, t-butylhydroquinone, 2,5-di-tbutylhydroquinone, phenylhydroquinone, methoxyhydroquinone,
chlorohydroquinone, 4-methylresorcinol, 4-chlororesorcinol,
3,3'-dimethyl-4,4'-dihydroxydiphenyl, 1,4-cyclohexanediol,
1,3-cyclohexanediol, 1,4-cyclohexanedimethanol, 1,3cyclohexanedimethanol, ethylene glycol, 1,3-propanediol,
1,4-butanediol.

Examples of the aromatic hydroxyamine and aromatic diamine (4) include 4-aminophenol, N-methyl-4-aminophenol, 1,4-phenylenediamine, N-methyl-1,4-phenylenediamine, N,N'-dimethyl-1,4-phenylenediamine, 3-aminophenol, 3-methyl-4-aminophenol, 2-chloro-4-aminophenol, 4-amino-1-naphthol, 4-amino-4'-hydroxydiphenyl, 2,6-diaminonaphthalene, 4,4'-diaminodiphenylmethane.

In addition, a molecular weight adjusting agent may be added to the above components, if necessary. Examples of the molecular weight adjusting agent include monofunctional components such as benzoic acid, phenol and 4-phenylphenol.

Among the components described above, more preferably examples are those which contain as essential components one or more kinds of compounds selected from naphthalene compounds, biphenyl compounds and phenylene compounds.

Typical examples of such liquid crystal polymers include the followings:

In the present invention, the ratio of the thermoplactic resin (A) not forming an anisotropic molten phase to the liquid crystal polymer (B) [(A)/(B)] is 99/1 to 50/50, preferably 99/1 to 80/20. When (A)/(B) is larger than 99/1, the fiberized LCP does not exhibit substantial reinforcing effects and an advantage cannot be obtained over the properties of the thermoplactic resin (A) contained only. On the other hand, when (A)/(B) is smaller than 50/50, since the matrix is the liquid crystal polymer, the properties of the thermoplastic polyester resin cannot be utilized

undesirably.

The effect of blending the compound (C) of the present invention is in that the compound (C) is highly effective as a dispersing assistant for micro-dispersing LCP (B) in the matrix phase and that the mechanical properties of the product are distinctively improved. In addition, this effect is stable as compared with that of the conventional phosphorus compound (triester phosphite).

The phosphorus oxoacid monoesters and diesters (C) used in the present invention are represented by the following formulae (I) and (II):

$$(X)_{n}P(=O)(OR)_{3-n}$$
 (I)

$$(X)_{n}P(OR)_{3\cdot n}$$
 (II)

wherein n is 1 or 2; X is a hydrogen atom, a hydroxyl group or a monovalent organic group and, when there are plural X's, they may be the same or different; and R is a monovalent organic group and, when there are plural R's, they may be the same or different.

Generally, phosphonate compounds, phophinate compounds, phosphonite compounds, phosphinite compounds and organic phosphorus compounds containing these phosphorus components in the molecule are applicable.

Specific examples of the phosphonate compound include dimethylphosphonate, diethylphosphonate, dibutylphosphonate, di(ethylhexyl)phophonate, didecylphosphonate,

dipalmitylphosphonate, distearylphosphonate,
dilaurylphosphonate, diphenylphosphonate,
dibenzylphosphonate, ditoluylphosphonate,
di (nonylphenyl)phosphonate, dioleylphosphonate,
dimethylmethylphosphonate, diethylmethylphosphonate,
di (ethylhexyl)methylphosphonate, dipalmitylmethylphosphonate,
distearylmethylphosphonate, dilaurylmethylphosphonate,
diphenylmethylphosphonate, dimethylphenylphosphonate,
diethylphenylphosphonate, di (ethylhexyl)phenylphosphonate,
dipalmitylphenylphosphonate, distearylphenylphosphonate,
dilaurylphenylphosphonate, diphenylphosphonate, and
dibenzylphenylphosphonate.

Specific examples of the phosphinate compound include methylphosphinate, ethylphosphinate, butylphosphinate, ethylphosphinate, palmitylphosphinate, stearylphosphinate, laurylphosphinate, phenylphosphinate, benzylphosphinate, toluylphosphinate, nonylphenylphosphinate, oleylphosphinate, ethylmethylphophinate, ethyldimethylphosphinate, ethyldimethylphosphinate, (ethylhexyl)methylphosphinate, (ethylhexyl)dimethylphosphinate, palmityldimethylphosphinate, stearylmethylphosphinate, stearyldimethylphosphinate, lauryldimethylphosphinate, phenylmethylphosphinate, ethylphenylphosphinate,

(ethylhexyl)phenylphosphinate, palmitylphenylphosphinate, stearylphenylphosphinate, stearyldiphenylphosphinate, laurylphenylphosphinate, phenylphenylphosphinate, and benzylphenylphosphinate.

Specific examples of the phosphonite compound include dimethylphosphonite, diethylphosphonite, dibutylphosphonite, di (ethylhexyl)phosphonite, didecylphosphonite, dipalmitylphosphonite, distearylphosphonite, dipalmitylphosphonite, diphenylphosphonite, dibenzylphosphonite, ditoluylphosphonite, di (nonylphenyl)phosphonite, dioleylphosphonite, dimethylmethylphosphonite, diethylmethylphosphonite, di (ethylhexyl)methylphosphonite, dipalmitylmethylphosphonite, distearylmethylphosphonite, dilaurylmethylphosphonite, diphenylmethylphosphonite, dimethylphenylphosphonite, diethylphenylphosphonite, dipalmitylphenylphosphonite, dipalmitylphenylphosphonite, distearylphenylphosphonite, dipalmitylphenylphosphonite, diphenylphosphonite, and dibenzylphenylphosphonite.

Specific examples of the phosphinite compound include methylphosphinite, ethylphosphinite, butylphosphinite, ethylphosphinite, ethylphosphinite, palmitylphosphinite, stearylphosphinite, laurylphosphinite, phenylphosphinite, benzylphosphinite, toluylphosphinite, nonylphenylphosphinite, oleylphosphinite, ethylmethylphosphinite,

ethydimethylphosphinite, (ethylhexyl)methylphosphinite,
(ethylhexyl)dimethylphosphinite, palmitylmethylphosphinite,
palmityldimethylphosphinite, stearylmethylphosphinite,
stearyldimethylphosphinite, laurylmethylphosphinite,
lauryldimethylphosphinite, phenylmethylphosphinite,
ethylphenylphosphinite, (ethylhexyl)phenylphosphinite,
palmitylphenylphosphinite, stearylphenylphosphinite,
stearyldiphenylphosphinite, laurylphenylphosphinite,
lauryldiphenylphosphinite, phenylphenylphosphinite, and
benzylphenylphosphinite. Among them, the phosphonic acid
ester represented by the formula (III) is especially
preferable:

$$H(OH)_{m}P(=O)(OR)_{2-m}$$
 (III)

wherein m is 0 or 1 and R is a monovalent organic group.

As the phosphorus compound (C), an organic phosphorus compound which contains the aforementioned components such as phosphonates, phophinates, phosphonites and phosphinites in the molecule thereof may also be used. Specific examples include the followings:

$$(XII)$$

$$(XII)$$

$$(XIII)$$

$$(XIII)$$

$$(XIII)$$

$$(XIII)$$

The amount of the specific phosphorus compound to be blended in the present invention is preferably 0.001-2.0 parts by weight, and more preferably 0.01-0.5 part by weight to 100 parts by weight in total of the components (A) and (B). When the amount is less than 0.001 part by weight, the effect of modifying the liquid crystal polymer into a fibrous state during the molding process is small. When the amount exceeds 2.0 parts by weight, the physical properties of the material may deteriorate.

Further, a thermoplastic resin which does not form an anisotropic molten phase other than the compounds (A) and (B) may be blended in an amount of 1-90 parts by weight to

100 parts by weight in total of the compounds (A) and (B) in the present invention. Examples of such an additional thermoplastic resin include polyethylene, polypropylene, ethylene-propylene copolymer, ethylene-vinyl acetate copolymer, polyamide, polyacetal, stylene-based resins such as polystylene, stylene-butadiene copolymer, stylenebutadiene-acrylonitrile copolymer and stylene-acrylonitrile copolymer, polyurethane, fluororesin, polyphenyleneoxide, polyphenylenesulfide, polybutadiene, butyl rubber, silicone resin, olefin-based thermoplastic elastomer, stylene-based thermoplastic elastomer, urethane-based thermoplastic elastomer, polyester-based thermoplastic elastomer, polyamide-based thermoplastic elastomer, polyether-based thermoplastic elastomer, multi-layer graft copolymer mainly containing polyacrylate and modified compounds thereof. Among them, preferred are stylene-based resin and polyphenyleneoxide which show relatively good mutual solubility to a polyester resin.

Furthermore, it is desirable to blend one or more kinds of silane compounds selected from vinylalkoxysilane, aminoalkoxysilane and mercaptoalkoxysilane in the present invention. By blending this component, an injection-molded article that shows little peeling on the surface thereof can be obtained.

Additionally, the molded article of the blend

composition of the present invention can be blended with various types of fibrous, powder and plate inorganic fillers according to the purposes of application. Examples of fibrous fillers include inorganic fibrous substances such as glass fibers, asbestos fibers, silica fibers, silica/alumina fibers, alumina fibers, zirconia fibers, boron nitride fibers, silicon nitride fibers, boron fibers, potassium titanate fibers, and further, fibrous metals, e.g., stainless, aluminum, titanate, copper and brass. Examples of powdery fillers include silicate such as carbon black, graphite, silica, quartz powders, glass beads, milled glass fibers, glass balloons, glass powders, calcium silicate, aluminum silicate, kaoline, talc, clay, diatom earth and wollastonite, metal oxide such as iron oxide, titanium oxide, zinc oxide, antinomy trioxide and alumina, metal carbonate such as calcium carbonate and magnesium carbonate, metal sulfate such as calcium sulfate and barium sulfate, and further, ferrite, silicon carbide, silicon nitride, boron nitride, and other various metal powders. Examples of tabular fillers include mica, glass flakes, various metal foils and the like. One or two or more types of these inorganic fillers can be used together. Moreover, the inorganic fillers used in the present invention can be used in combination with a known surface treatment agent in accordance with desired physical properties. For example,

functional compounds such as epoxy compounds, isocyanate compounds, titanate compounds and silane compounds are cited. Preferably, treatment is performed with the compounds other than amino compounds, such as epoxy compounds and polyamide compounds. These fillers may be used after surface treatment, or added at the same time of material preparation. Herein, the amount of the inorganic fillers to be blended is preferably 1 to 100 parts by weight based on 100 parts by weight of the total amount of (A) and (B).

Moreover, the molded article may be flame-retarded by blending a flame retardant. As the flame retardant, organic halogen compounds and the like are used, but especially aromatic bromine compounds are preferable. Additionally, as a flame retardant assistant, metal oxide and hydroxide such as antimony trioxide, antimony halide, aluminum hydroxide and magnesium hydroxide are preferably used.

In this resin composition, LCP is preferably present as fibers having an average aspect ratio of 5 or more inside the molded article, especially preferably having the average aspect ratio of 8 or more. When the average aspect ratio is less than 5, a high strength and a high elasticity modulus as the characteristics of this resin composition cannot be obtained, and especially, a thin-film molded article for miniaturization and lightening cannot obtain a sufficient rigidity.

•

The composition of the present invention can generally be prepared by the equipment and methods used in preparing a synthetic resin composition. For example, there is a method of mixing necessary components, using a uniaxial or twinscrew extruder, kneading/extruding to prepare molding pellets and, subsequently, molding; a method of once preparing pellets having different compositions from one another, mixing a specific amount of the pellets to be subjected to molding, and obtaining a molded article having an intended composition; or the like. Namely, any method of preparing the composition before molding may be used, since LCP is formed into fibers during molding in the present invention. Additionally, LCP contained in the composition is preferably molded at a temperature at which LCP flows by a shearing force during molding, especially at a temperature of a melting point of LCP or higher. When the temperature is the LCP's melting point or higher, the LCP's property of easy flow by the shearing force is utilized, and LCP can easily be formed into fibers by the shearing force.

Examples

Hereinafter, the present invention will be described in more detail with reference to the following examples, but the present invention is not limited to the examples.

Example 1

0.1 part by weight of dioleylphosphonate (manufactured by Johoku Kagaku Kogyo Co., Ltd.) was added to a resin component containing 80 parts by weight of a polycarbonate resin (Upiron S3000, manufactured by Mitsubishi Gas Chemical Co., Ltd.) and 20 parts by weight of a liquid crystal polymer (A950, manufactured by Polyplastics Co., Ltd.). The mixture was melt-kneaded at a resin temperature 290 °C in a 30 mm twin-screw extruder, and pelletized to obtain the aimed resin composition.

In the extrusion process, the aimed resin composition was prepared with removing the volatile components via a vent by a pressure-reducing operation, while care was taken so that air should not be mixed into the kneaded materials and oxidize them.

Then, the obtained pellets were formed by injection molding into a test piece having thickness of 0.8 mm, width of 12.7 mm and length of 131 mm at a resin temperature 290 $^{\circ}\text{C}$.

The bending strength and flexural modulus of the test piece were measured according to ASTM D 790.

The test piece was cut in the flow direction and the surface of the section was mirror-polished. When the polished surface was observed by an electronic microscope to evaluate the condition of the surface, it was observed that

the liquid crystal polymer formed a fibrous structure in the polycarbonate resin. 50 fibers of the liquid crystal polymer in a fibrous state were arbitrarily chosen and thickness/diameter thereof were measured to calculate the average values of thickness/diameter. With respect to the length of the fibers, the length which could be observed on

Comparative Example 1

the surface was measured.

A test piece was molded and physical properties thereof were evaluated in the same manner as Example 1 except that dioleylphosphonate was not added. Further, the test piece was cut in the flow direction and the surface of the section was mirror-polished. When the polished surface was observed by an electronic microscope to evaluate the condition of the surface, a structure was observed in which the liquid crystal polymer was dispersed insularly in the polycarbonate resin.

Examples 2-5, Comparative Examples 2-3

A test piece was molded and physical properties thereof were evaluated in the same manner as Example 1 except that a phosphorus compound shown in Table 1 was added in an amount shown in Table 1 in place of dioleylphosphonate.

Example 6

A test piece was molded and physical properties
thereof were evaluated in the same manner as Example 1
except that the blending ratio of the polycarbonate resin to
the liquid crystal polymer was changed as shown in Table 1.

Example 7

A test piece was molded and physical properties thereof were evaluated in the same manner as Example 1 except that the polyarylate resin (U-Polymer, U-1000 manufactured by Unichika Co., Ltd.) was used in place of the polycarbonate resin.

Comparative Example 4

A test piece was molded and physical properties thereof were evaluated in the same manner as Example 7 except that dioleylphosphonate was not added.

Example 8

0.1 part by weight of dioleylphosphonate and 30 parts by weight of glass fiber (chopped strand having a fiber diameter of 13 μ m and a fiber length of 5 mm) were added to a resin component containing 50 parts by weight of the polycarbonate resin and 20 parts by weight of the liquid crystal polymer. Then, a test piece was molded and physical

properties thereof were evaluated in the same manner as other Examples.

Example 9

0.1 part by weight of dioleylphosphonate, 30 parts by weight of glass fiber (chopped strand having a fiber diameter of 13 µm and a fiber length of 5 mm) and 10 parts by weight of fine mica having the average particle diameter of 18 µm and the average aspect ratio of 20 were added to a resin component containing 40 parts by weight of the polycarbonate resin and 20 parts by weight of the liquid crystal polymer. Then, a test piece was molded and physical properties thereof were evaluated in the same manner as other Examples (except that a 44 mm twin-screw extruder was used).

Comparative Example 5

A test piece was molded and physical properties thereof were evaluated in the same manner as Example 9 except that dioleylphosphonate was not added. In this test piece, the dispersed state of the liquid crystal copolymer was not observed.

The results of evaluation described above are shown in Table 1.

Table 1

Com. Ex. 5 40	30	10	123	11000		
	3	-	15			
Com. Ex. 4 80 20 20			120	2780	insula	1.1
Com. Ex. 3 80 20 20	0.1		93	2850	insular	1.3
Com. Ex. 2 80 20 20 20 0.1			110	4500	fibrous	4.2
Com. Ex. 1 80 80 20 20			95	2880	insular	1.5
Ex. 9 40 20 20 0.1	30	10	224	16300	fibrous	>9.0
Ex. 8 50 50 0.1	30		240	14900	fibrous	>9.0
Ex. 7 80 20 20 0.1			195	6550	fibrous insular fibrous insular insular	5.8
Ex. 6 60 60 40 0.1			168	8350	fibrous	6.4
Ex. 5 80 80 20 20			135	5800	fibrous	6.5
Ex. 4 80 20 20 0.1			143	6100	fibrous	8.6
Ex. 3 80 80 20 0.3			138	6010	fibrous	7.3
Ex. 2 80 80 20 20 0.1			140	0809	fibrous	7.2
Ex. 1 80 80 20 0.1			145	0209	fibrous	9.0
(A) Polycarbonate resin (A) Polyarylate resin (B) Liquid crystal polymer (A950) Dioleylphosphonate Dioctadecylphosphonate Dinonylphenylphosphonate (C) Mixture of monostearylphophate/ destearylphosfate in 44/55 Bis(2,6-di-t-butyl-4-metylphenyl) pentaerythritoldiphosphite	Tridecylphosphate s fiber		Bending strength MPa	Flexial modulus MPa	Dispersing state of liquid crystal polymer	Average aspect ratio
(A) Polyce (A) Polyce (B) Liquit (B) Liquit (C) Mixt deste (C) Mixt deste (C) Mixt	Tridec Glass fiber	Mica	}endi	'lexia	ispe f liqu	rver.

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Claims

1. A thermoplastic resin composition comprising 100 parts by weight in total consisting of 99 to 50 parts by weight of a thermoplastic polyester resin (A) which does not form an anisotropic molten phase and 1 to 50 parts by weight of a liquid crystal polymer (B) capable of forming an anisotropic molten phase, and 0.001 to 2.0 parts by weight of one or more kinds of compounds (C) selected from phosphorus oxoacid monoesters and diesters represented by the following formulae (I) and (II):

$$(X)_{n}P(=O)(OR)_{3-n}$$
 (I)

$$(X)_{n}P(OR)_{3,n} \qquad (II)$$

wherein n is 1 or 2; X is a hydrogen atom, a hydroxyl group or a monovalent organic group and, when there are plural X's, they may be the same or different; and R is a monovalent organic group and, when there are plural R's, they may be the same or different.

- 2. The composition according to claim 1, wherein the thermoplastic polyester resin (A) not forming an anisotropic molten phase includes one or more kinds of resins selected from polycarbonate resin, polyalkylene terephthalate resin and polyarylate resin.
- 3. The composition according to claim 1, wherein the thermoplastic polyester resin (A) not forming an anisotropic

molten phase includes one or more kinds of resins selected from polycarbonate resin and polyarylate resin.

- 4. The composition according to claim 1, wherein the thermoplastic polyester resin (A) not forming an anisotropic molten phase is polycarbonate resin.
- 5. The composition according to claim 2, wherein the polyalkylene terephthalate resin includes polyethylene terephthalate and/or polybutylene terephthalate resin.
- 6. The composition according to any of claims 1 to 4, wherein the phosphorus oxoacid monoester and diester (C) are phosphonates represented by the following formula (III):

$$H(OH)_{m}P(=O)(OR)_{2-m}$$
 (III)

wherein m is 0 or 1 and R is a monovalent organic group.

7. The composition according to any of claims 1 to 5, wherein the phosphorus oxoacid monoester and diester (C) are phosphonates represented by the following formula (IV):

$$HP(=O)-Y \qquad (IV)$$

wherein Y is a divalent α , ω -dioxy organic group.

8. The composition according to any of claims 1 to 7, which further contains a thermoplastic resin not forming an anisotropic molten phase, except for (A) and (B), in an amount of 1 to 90 parts by weight to 100 parts by weight of the total amount of (A) and (B).

- 9. The composition according to any of claims 1 to 8, which further contains an inorganic filler in an amount of 1 to 100 parts by weight to 100 parts by weight of the total amount of (A) and (B).
- 10. A molded article prepared by molding the composition according to any of claims 1 to 9.
- 11. An injection molded article, wherein the liquid crystal polymer (B) capable of forming an anisotropic molten phase is present in the state of fibers having an average aspect ratio of 5 or more in a matrix of the thermoplastic polyester resin (A) not forming an anisotropic molten phase as a result of an injection molding of the composition according to any of claims 1 to 9.

Abstract

To provide a resin composition, in which a liquid crystal polymer is made into fiber in a molded article to exhibit an extremely high reinforcing effect which has not been available yet, and from which a molded article having an excellent mechanical strength can be manufactured in a stable manner. That is, a thermoplastic resin composition, wherein 100 parts by weight in total consisting of 99-50 parts by weight of a thermoplastic polyester resin (A) not forming an anisotropic molten phase and 1-50 parts by weight of a liquid crystal polymer (B) capable of forming an anisotropic molten phase is blended with 0.001-2.0 parts by weight of one or more kinds of compounds (C) selected from phosphorus oxoacid monoester and diester represented by the following formulae (I) and (II):

$$(X)_{n}P(=O)(OR)_{3-n}$$
 (I)

$$(X)_{n}P(OR)_{3-n} \qquad (II)$$

wherein n is 1 or 2; X is a hydrogen atom, a hydroxyl group or a monovalent organic group and, when there are plural X's, they may be the same or different; and R is a monovalent organic group and, when there are plural R's, they may be the same or different.

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Japan	11-202839	16 July 1999	YES_X_NO
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Shizuoka, Japan		Japan			
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c/o Polyplastics Co., Ltd. 973, Miyajima, Fuji-shi, Shizuoka, Japan		•			
FULL NAME OF SECOND JOINT INVENTOR, IF ANY	SIGNATURE (DATE		
Toshio Shiwaku	4-11	1~~	Nov.	14,	2001
RESIDENCE		CITIZENSHIP			
Shizuoka, Japan		Japan			
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FULL NAME OF THIRD JOINT INVENTOR, IF ANY	SIGNATURE		DATE
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c/o Polyplastics Co., Ltd. 973, Miyajima, Fuji-shi, Shizuoka, Ja FULL NAME OF FOURTH JOINT INVENTOR, IF ANY	SIGNATURE		T
ODDINEND OF TOOMSTORY INVENTOR, IF ANY	SIGNATORE		DATE
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full name of fifth joint inventor, if any	SIGNATURE		DATE
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FULL NAME OF SIXTH JOINT INVENTOR, IF ANY	SIGNATURE		DATE
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FULL NAME OF SEVENTH JOINT INVENTOR, IF ANY	SIGNATURE		DATE
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FULL NAME OF EIGHTH JOINT INVENTOR, IF ANY	SIGNATURE		DATE
The state of brother beautiful and beautiful freeze	SIGNATURE		DATE
ESIDENCE		CITIZENSHIP	
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ULL NAME OF NINTH JOINT INVENTOR, IF ANY	SIGNATURE		DATE
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ULL NAME OF TENTH JOINT INVENTOR, IF ANY	SIGNATURE		DATE
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